

A major purpose of the Technical Information Center is to provide the broadest dissemination possible of information contained in DOE's Research and Development Reports to business, industry, the academic community, and federal, state and local governments.

Although a small portion of this report is not reproducible, it is being made available to expedite the availability of information on the research discussed herein.

TITLE COMPOSITE FOAMS

AUTHOR(S) Alice M. Nvitray, MST-7  
Joel M. Williams, MST-7SUBMITTED TO Advanced Materials Workshop  
Lawrence Livermore National Laboratory  
Livermore, California  
July 26-28, 1988**DISCLAIMER**

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

By acceptance of this article the publisher recognizes that the U.S. Government retains a nonexclusive, royalty-free license to publish or reproduce the published form of this contribution, or to allow others to do so, for U.S. Government purposes.

The Los Alamos National Laboratory requests that the publisher identify this article as work performed under the auspices of the U.S. Department of Energy.



**Los Alamos** Los Alamos National Laboratory  
Los Alamos, New Mexico 87545

## COMPOSITE FOAMS

Alice M. Nytray, Joel Williams and Mark Wilkerson  
Los Alamos National Laboratory  
Materials Science and Technology Division  
Los Alamos, New Mexico

### Introduction

Composite foams have been prepared which incorporate the best attributes of two materials. Polystyrene emulsion foams prepared by the Unilever method<sup>1</sup>, Si aerogel, and resole foams have been studied for use in laser fusion targets<sup>2</sup>. Foams for this application need to be strong enough to withstand machining operations and handling, yet they also require small pore/cell sizes because they must hold liquid DT against gravitational forces. These attributes are difficult to achieve in a single foam. The polystyrene emulsion foams have the advantages that they can be prepared easily over a wide range of densities (0.15 g/cc to 0.20 g/cc) and are easy to handle and machine, however, the small cell sizes needed for good wicking of the DT liquid fuel are difficult to achieve. On the other hand, Si aerogel and resole foams have the desired small cell size ( $< 0.1 \mu\text{m}$ ), but the materials are mechanically weak making them difficult to handle and machine. Using a backfilling process, we have been able to make binary composite foams in which the cells of the polystyrene emulsion foam have been filled with aerogel or resole foam. These materials have the small cell sizes needed for good wicking of the liquid DT fuel and are rugged and easy to machine.

Figure 1 shows the typical open cell structure of a polystyrene emulsion foam (0.1 g/cc) with a cell size of about  $30 \mu\text{m}$ . Figure 2 and 3 shows the same foam backfilled with Si aerogel at several different concentrations. Good filling was obtained down to aerogel concentrations of

around 25mg/cc. At 10 mg/cc there was insufficient Si aerogel to complete fill the polystyrene foam.

Figure 4 and 5 show the resole foams prepared from resorcinol-formaldehyde and phloroglucinol-formaldehyde respectively. Although these materials filled the cells, the composite foams tended to shrink somewhat upon extraction especially at the higher densities of resorcinol-formaldehyde. These same characteristics were reported earlier for the resole foams<sup>2</sup> The phloroglucinol-formaldehyde filled composite foams showed less shrinkage and could be successfully filled to lower concentrations.

Deuterium wicking experiments were performed at the National Bureau of Standards on several of the composite foams (Figure 6). The data (Fig 7-8) indicates that the aerogel filled composite foam as well as the low density P/F filled foam wicked the liquid deuterium better than any of the individual materials.<sup>3</sup>

Compression tests indicate that the strength of the composite foam as well as its machinability is determined solely by the strength of the polystyrene foam.

At the present time, we are working to broaden the range of composite foam densities and to determine both the chemical and physical properties of these new materials.

## **Experimental Section**

### **Preparation of the silica aerogel filled polystyrene emulsion foams.**

Two stock solutions were prepared: a 250 ml stock solution containing 130.4g of tetramethyloxosilicate in anhydrous methanol and a 200 ml stock solution containing 48g of water in methanol. Equal amounts of solution of the stock solutions were mixed and diluted with additional methanol (or methanol and toluene) to give the desired density of filler. For example, 100 mg/cm<sup>3</sup> silica filler (Figure 1) was prepared by mixing 20 ml of each stock solutions with 10 ml of toluene. The polystyrene foam was immersed in the solution and trapped air was removed from the foam by drawing a vacuum over the solution. After the foam was saturated with the solution three drops of catalyst (either fluoroboric acid or concentrated ammonium hydroxide) were added to the solution to initiate the polymerization. Generally, the gelation occurred within 24h. The

excess gel was removed from the surface of the foam; and, the foam was placed in a Polaron to remove the solvents from the gel network. After a week of liquid CO<sub>2</sub> extraction, the carbon dioxide was supercritically extracted by raising the temperature of the Polaron to 40C and 1400psi. The CO<sub>2</sub> gas was then vented slowly overnight.

#### Preparation of Resorcinol-formaldehyde filled polystyrene emulsion foam.

To make a 6 wt% resorcinol-formaldehyde foam, 3.25 g resorcinol and 0.05g sodium carbonate were dissolved in 50g of distilled water. While stirring, 25 ml of isopropyl alcohol and 4.80g of 37% formaldehyde were added. The solution was placed into a bottle. The polystyrene foam was immersed in the solution and trapped air was removed from the foam by drawing a vacuum over the solution. The bottle was capped and placed in a 70C oven for a week to cure. The reddish-amber gel was removed from the exterior of the foam and the solvent exchange process was begun. First, the filled foam was placed in a 5% acetic acid solution at 50C for 24h. The acid solution was then replaced by methanol. The methanol was replaced with fresh methanol every day for at least one week. The foam was placed in a Polaron to remove the solvents from the gel network. After a week of liquid CO<sub>2</sub> extraction, the carbon dioxide was supercritically extracted by raising the temperature of the Polaron to 40C and 1400psi. The CO<sub>2</sub> gas was then vented slowly overnight.

#### Phloroglucinol-formaldehyde filled polystyrene emulsion foam.

The phloroglucinol-formaldehyde filled foams were prepared in the same manner as the resorcinol-formaldehyde foams except phloroglucinol was used in place of resorcinol.

## References

1. Unilever Research Laboratory, *Emulsion Process for Making Foams*, European Patent 60138 (Sept. 3, 1982).
2. Progress Report 1986, *Low-Density Hydrocarbon Foams for Laser Fusion Targets*, Lawrence Livermore National Laboratory, Livermore, Calif., UCID-21080-86.
3. Proceedings of the Fifth Target Fabrication Specialist Meeting, 1986, Las Vegas, Nevada, Conf-860330, Vol. 1, p. 159.

## **List of Figures**

**Figure 1.** SEMS of Polystyrene Emulsion Foam

**Figure 2.** SEMS of Si Aerogel/Polystyrene Composite Foam

**Figure 3.** SEMS of Si Aerogel/Polystyrene Composite Foam

**Figure 4.** SEMS of Resorcinol-Formaldehyde/Polystyrene Composite Foams

**Figure 5.** SEMS of Phloroglucinol-Formaldehyde/Polystyrene Composite Foams

**Figure 6.** Liquid Deuterium wicking data

**Figure 7.** Liquid Deuterium wicking data

**Figure 8.** Liquid Deuterium wicking data

Polystyrene Emulsion Foam  
Density = 0.1g/cc

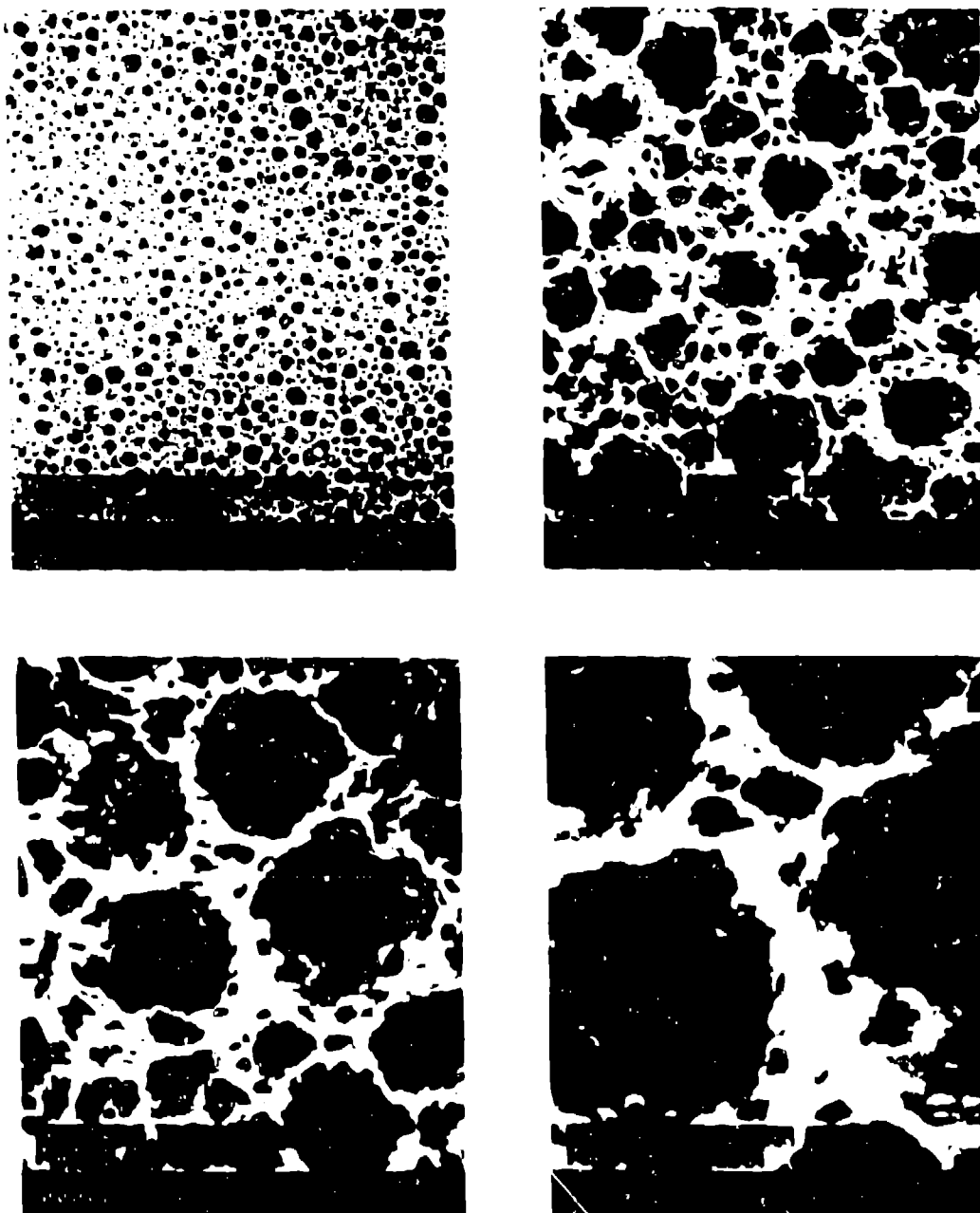


Figure 1



## Si Aerogel Foam in Polystyrene Foam

100 mg/cc SiO foam  
in 0.1 g/cc PS foam



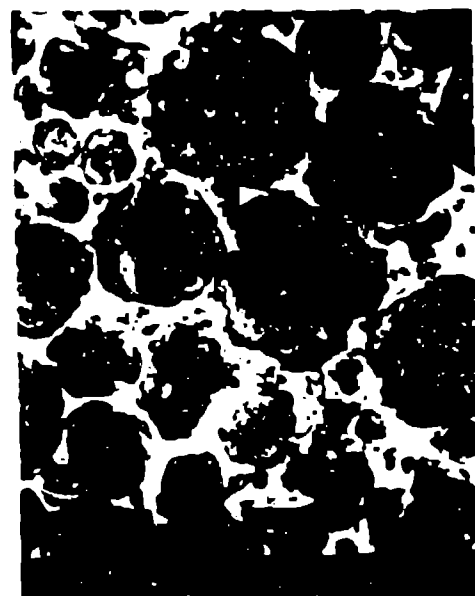
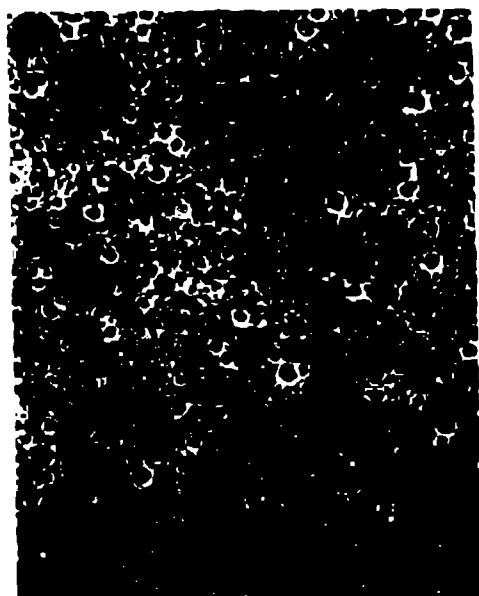
50 mg/cc SiO foam  
in 0.1 g/cc PS foam



Figure 2

## Si Aerogel Foam in Polystyrene Foam

25 mg/cc SiO foam  
in 0.1 g/cc PS foam



10 mg/cc SiO foam  
in 0.1 g/cc PS foam

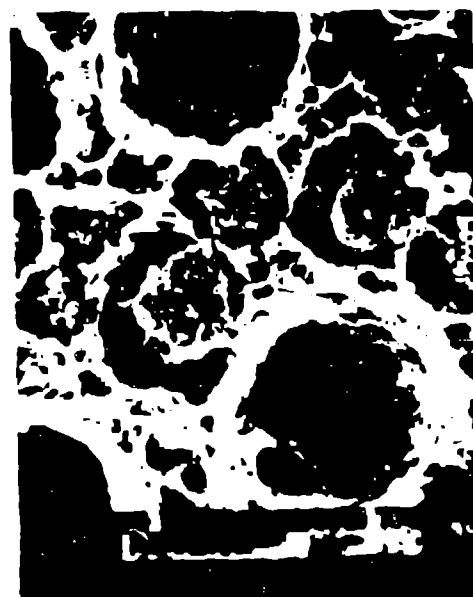
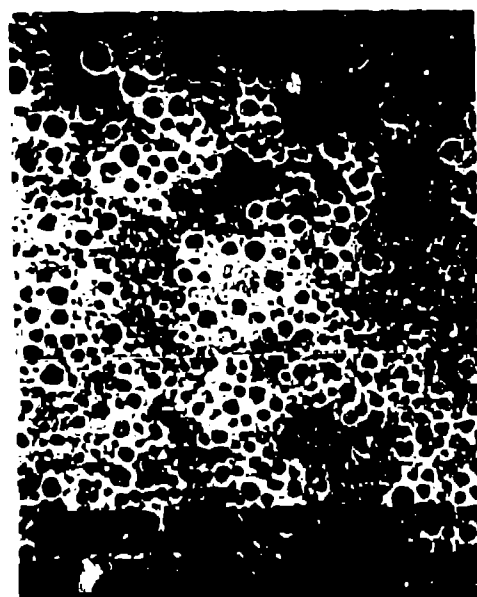
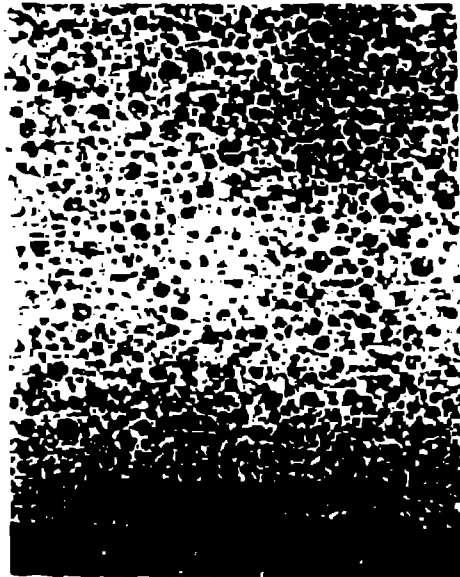


Figure 3

## Resorcinol-Formaldehyde Foam in Polystyrene Foam

6 wt% R/F foam

in 0.1 g/cc PS foam



4 wt% R/F foam

in 0.1 g/cc PS foam



Figure 4

## Phloroglucinol-Formaldehyde Foam in Polystyrene Foam

7 wt% P/F foam  
in 0.1 g/cc PS foam



2 wt% P/F foam  
in 0.1 g/cc PS foam

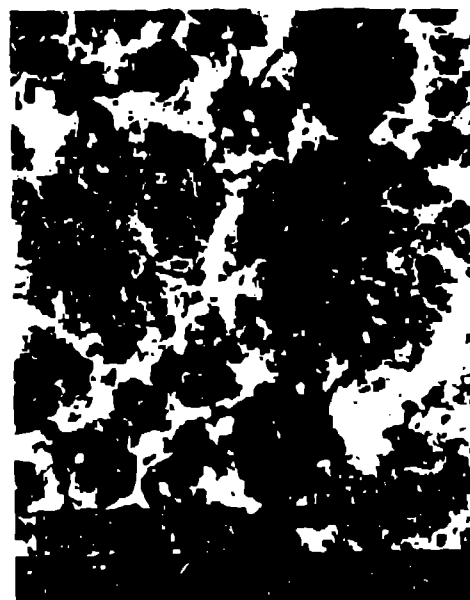
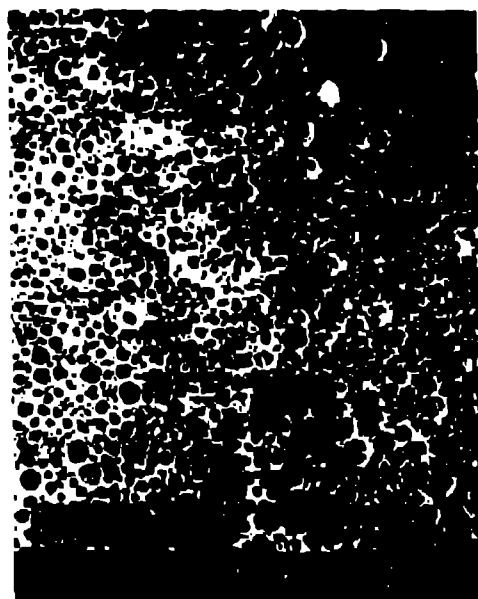


Figure 5

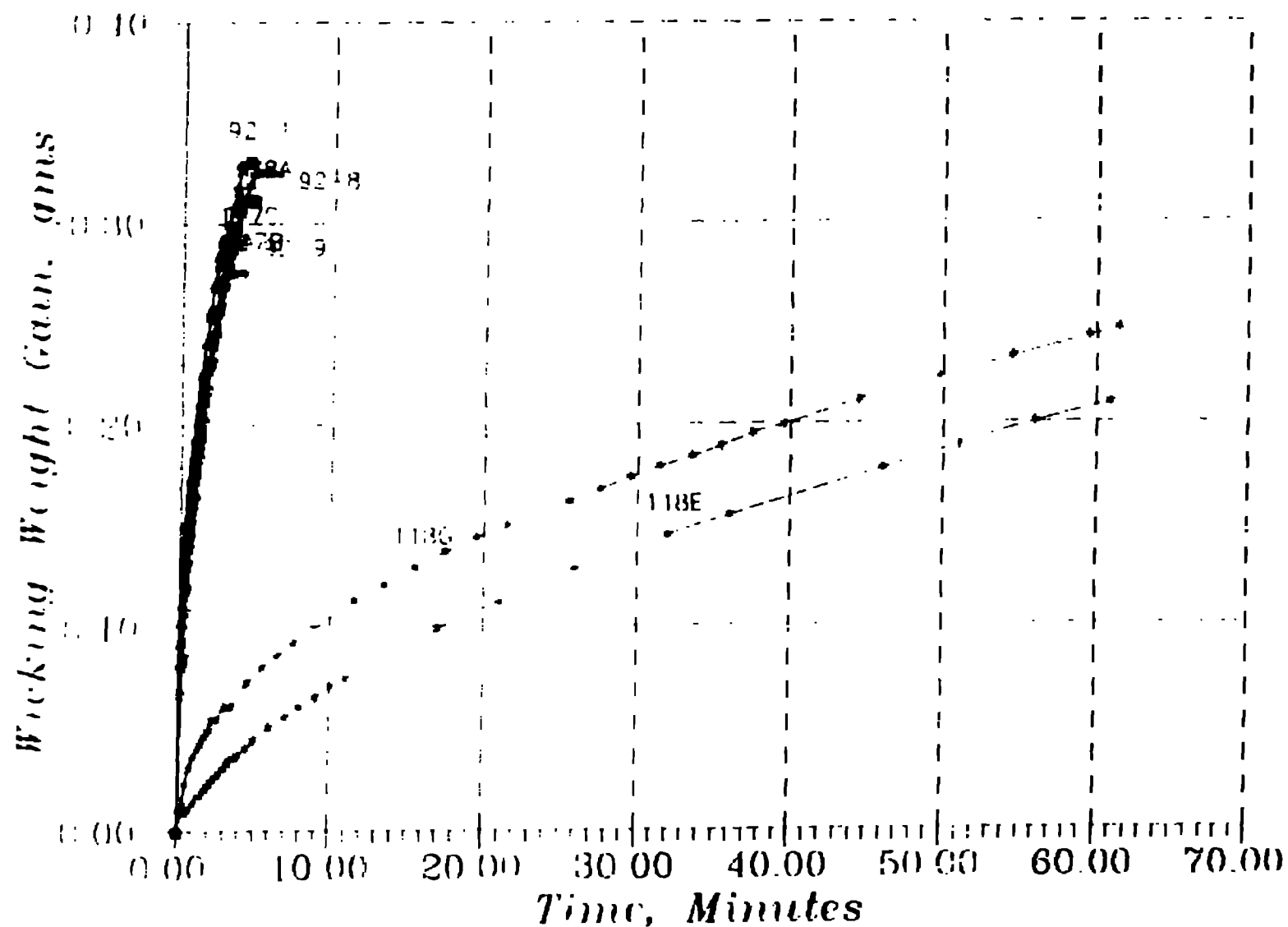


Figure 6

		<u>Length/diameter (ci)</u>
92-1	Aerogel (100mg/cc)-PS	6.376/0.647
92-2	Aerogel (50mg/cc)-PS	6.366/0.642
92-3	Aerogel (25mg/cc)-PS	5.772/0.640
118A	Phloroglucinol/Formaldehyde(2 wt%)-PS	6.292/0.633
118B	Phloroglucinol/Formaldehyde(6 wt%)-PS	6.498/0.602
118C	Phloroglucinol/Formaldehyde(4 wt%)-PS	6.504/0.606
118D	Resorcinol/Formaldehyde(4 wt%)-PS	6.313/0.641
118E	Resorcinol/Formaldehyde(5 wt%)-PS	6.280/0.638



Figure 9

Composite Foam	Foam Density (g/cc)	D2 (g)	D2 (cc)	Porosity %
Aerogel/PS (100mg/cc)	0.1121 (0.235/2.096)	0.312	1.518	86.73
Aerogel/PS (50mg/cc)	0.1203 (0.248/2.061)	0.31	1.564	80.46
Aerogel/PS (25mg/cc)	0.0996 (0.185/1.857)	0.270	1.573	84.70
5wt% PF/PS	0.1681 (0.311/1.850)	0.262	1.523	82.34
4 wt% PF/PS	0.1407 (0.264/1.876)	0.271	1.579	84.16
2 wt% PF/PS	0.1199 (0.237/1.977)	0.293	1.707	86.35
5 wt% RF/PS	0.1444 (0.288/1.995)	0.296	1.724	86.44
4 wt% RF/PS	0.1514 (0.303/1.967)	0.271	1.576	82.58